

Qualitative Chemistry: Precipitation of Cations and Anions

Introduction

Qualitative chemistry is the subdivision of chemistry concerned with identifying substances. By understanding how different substances react with solvents and with each other, it is possible to deduce their identity by performing systematic tests on them. These tests might include: 1) solubility tests to see if a solid dissolves in a solvent, 2) precipitation tests to see if a solid is formed when two dissolved substances are mixed, 3) pH tests to determine the concentration of dissociated hydrogen ions (H^+). All of these tests will produce information about the properties of the unknown substance and aid in its identification.

Solubility and Precipitation Tests

For the experiments you will perform today, water will be the only solvent utilized. Thus, it will be important to understand the rules for solubility of salts in water. They are:

1. Most nitrate (NO_3^-) salts are soluble.
2. Most salts containing the alkali metal ions (Li^+ , Na^+ , K^+ , Rb^+ , Cs^+) and the ammonium ion (NH_4^+) are soluble.
3. Most chloride (Cl^-), bromide (Br^-) and iodide (I^-) salts are soluble. Notable exceptions are salts containing the ions Ag^+ , Pb^{2+} and Hg_2^{2+} .
4. Most sulfate (SO_4^{2-}) salts are soluble. Notable exceptions are BaSO_4 , PbSO_4 , HgSO_4 and CaSO_4 .
5. Most hydroxide salts are only slightly soluble. The important soluble hydroxides are NaOH and KOH . The compounds $\text{Ba}(\text{OH})_2$, $\text{Sr}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$ are marginally soluble.
6. Most sulfide (S^{2-}), carbonate (CO_3^{2-}), chromate (CrO_4^{2-}) and phosphate (PO_4^{3-}) salts are only slightly soluble.

With the aid of these solubility rules, you can **predict if a solid will dissolve in water and if a solid will be formed when two soluble salts are combined in solution**. For example, if you mixed solutions of KI and Pb(NO₃)₂ together, would a solid be formed? The two product compounds to consider are KNO₃ and PbI₂. From solubility rule #1 we can deduce that KNO₃ will dissolve. However, solubility rule 3 clearly states that PbI₂ is a notable exception to I⁻ salts being soluble. Therefore we can deduce that PbI₂ solid will be formed when KI(aq) and Pb(NO₃)₂(aq) are mixed together.

For this experiment, you are only permitted to **use the eight unknown solutions on each other**. No other solutions should be mixed with them. All of the solutions are aqueous. The possible species present are: K⁺, Ag⁺, Ca²⁺, Cu²⁺, Pb²⁺, NO₃⁻, SO₄²⁻, PO₄³⁻, Cl⁻, I⁻, OH⁻ and H₂O (i.e., one of the solutions could be pure water!). Remember that ionic compounds consist of a cation and anion. In the reactions either of the species could be precipitating (causing the precipitation).

The following information will assist you identifying the salts contained in the eight unknown solutions:

1. The solutions have not been doctored to deceive you. (no additional colors or scents)
2. Check the pH of all the solutions with pH paper first. OH⁻ and PO₄³⁻ will form basic solutions.
3. After mixing the solutions, work on the unique precipitates first since they will be easiest to identify.
4. **Each solution contains one cation and one anion** but a specific ion can occur in more than one solution. (Disregard H⁺ and OH⁻ from water auto-ionization). For example 2 solutions may have K⁺ ions.
5. All the reactions that will occur are precipitation and complexation reactions except one redox reaction: $2\text{Cu}^{2+} + 4\text{I}^{-} \rightarrow 2\text{CuI}(\text{s}) + \text{I}_2$ (The brownish white CuI and the brown I₂ will appear together as a brown-rust color.)

6. The following precipitates might occur:

Precipitate	Color	Precipitate	Color
PbCl_2	white	$\text{Cu}_3(\text{PO}_4)_2 \cdot 3\text{H}_2\text{O}$	bluish-white
PbI_2	yellow	$\text{Cu}(\text{OH})_2$	blue
$\text{Pb}(\text{OH})_2$	white	CuI	brown
PbSO_4	white	Ag_2O	brown, from Ag^+ and OH^-
$\text{Pb}_3(\text{PO}_4)_2$	white	AgCl	white
$\text{Ca}(\text{OH})_2$	white	Ag_3PO_4	yellow
CaSO_4	white	AgI	yellow

Equipment needed

2 plastic coated reaction grids
8 disposable pipets
pH paper

Procedure

Label eight of your small test tubes from 1 to 8 (it is OK to use 2 large test tubes). Take your labeled test tubes to the hood and add **one or two milliliters** of the unknown solution of corresponding number (you don't need much). Obtain 8 disposable pipets and place one in or beside each test tube. Be careful not to mix up your pipets!

Obtain some pH paper. Place a small drop of each solution on the pH paper. You do not need a full strip for each solution. Record the approximate pH of each solution in your lab notebook. pH significantly above 7 indicate a basic solution, pH significantly below 7 indicate a acidic solution.

The next step will be done twice (one time for the known solutions and one time for the unknowns) using two separate plastic coated grids.

Notice that each box in the **plastic coated reaction grid** is half white and half black. The black section will aid in identifying white precipitates. Therefore, be sure to place drops so that they will be over both the white and black sections. Place **one or two drops** of the first solution on every square on the first column of the grid. Slowly add **one to two drops** of the second solution to the first solution in the square corresponding to (1,2) or (2,1). Continue to place drops of solution 2 in every square of the second column. Repeat for the remaining six solutions in this way until the entire grid is complete. Record your observations in the data chart of the Data Sheet, noting the color and amount of any precipitate (for example: slight amount white precipitate).

Compare the two reaction grids: this will help in identifying the unknown solutions.

As you identify the cation and anion of each solution, record them in your data chart of the Data Sheet. Also record the results of your pH and flame tests in the same Data Sheet.

Wipe your plastic coated grids with paper towel to clean them. Please return any hood items for the next class' use. Do not pour lead-containing solutions down the sink! Please dispose of these in the waste bottle provided for you in the hood. If you are uncertain if a solution contains lead, better safe than sorry- use the waste bottle.

Table 1 General Properties of Cations

Name	Formula	Color of Aqueous Soln	Flame Test Color	Color of Salts
1. copper(II) ion (cupric ion)	Cu^{2+}	blue or green	deep blue or green	depends on anion
2. lead(II) ion	Pb^{2+}	colorless	faint blue-gray	some colored
3. calcium(II) ion	Ca^{2+}	colorless	red-orange	colorless
4. potassium(I) ion	K^{+}	colorless	fleeting weak violet	colorless
5. silver(I) ion	Ag^{+}	colorless		depends on anion

NAME: _____

DATE: _____

SECTION: _____

Pre-lab Assignment: Qualitative Analysis

1. Please fill in the following chart with predicted **solid** reaction products for the series of anions and cations listed in this experiment. When reporting these products, be sure to include their correct **chemical formula** and the **color** that you would expect to observe upon solid formation. Indicate by **NR** that no detectable reaction or precipitate will be formed. You may want to refer to page 3 of this handout for help identifying colors of precipitates. You should use the solubility rules given on page 1.

	OH^-	Cl^-	I^-	NO_3^-	SO_4^{2-}	PO_4^{3-}
K^+						
Ag^+						
Ca^{2+}						
Cu^{2+}						
Pb^{2+}						

2. You are given four unlabeled bottles, each containing a clear liquid, and the following information:

- In the 4 bottles Ag^+ , H^+ (ignore the dissociation of water as a source of H^+), K^+ , Pb^{2+} , CrO_4^{2-} , NO_3^- , SO_4^{2-} and I^- are present.
- Each cation and anion are present in only one of the four bottles.
- The following salts are only sparingly soluble and form precipitates:
 Ag_2CrO_4 (red), AgI (yellow),
 PbCrO_4 (yellow), PbI_2 (yellow), PbSO_4 (white)
- Assume all other possible salts are soluble enough so that no precipitate will be detected.
- The following redox reaction is possible:
 $2\text{H}^+ + 2\text{CrO}_4^{2-} \rightarrow \text{H}_2\text{O} + \text{Cr}_2\text{O}_7^{2-}$
yellow solution \rightarrow orange solution

Tests were performed on this set of four unlabeled bottles as in this experiment and the following table was constructed:

	1		
2	white ppt		2
3	yellow ppt	red ppt	3
4	yellow ppt	yellow ppt	Yellow soln \rightarrow orange

Fill in the boxes with the chemical formulas of the precipitates.

Identify the cation and anion in each bottle:

Bottle #	1	2	3	4
Cation	_____	_____	_____	_____
Anion	_____	_____	_____	_____

NAME: _____

DATE: _____

PARTNER: _____

SECTION: _____

Data Sheet: Known Solution Analysis

1	Pb(NO ₃) ₂								
2	KOH	2	KOH						
3	AgNO ₃		3	AgNO ₃					
4	K ₃ PO ₄			4	K ₃ PO ₄				
5	CaCl ₂				5	CaCl ₂			
6	KI					6	KI		
7	CuSO ₄						7	CuSO ₄	
8	H ₂ O								

NAME: _____

DATE: _____

PARTNER: _____

SECTION: _____

Data Sheet: Unknown Solution Analysis

1								
2	2							
3		3						
4			4					
5				5				
6					6			
7						7		
8								

NAME: _____

DATE: _____

SECTION: _____

Post-lab Questions: Qualitative Analysis

Please fill in the following chart for the unknown solutions.

Remember that each solution contains one cation and one anion.

Solution #	pH	Cation	Anion	Compound
1	_____	_____	_____	_____
2	_____	_____	_____	_____
3	_____	_____	_____	_____
4	_____	_____	_____	_____
5	_____	_____	_____	_____
6	_____	_____	_____	_____
7	_____	_____	_____	_____
8	_____	_____	_____	_____